AD NUMBER
AD479698
LIMITATION CHANGES
TO: **Approved for public release; distribution is unlimited.**
FROM: **Distribution authorized to U.S. Gov't. agencies and their contractors; Critical Technology; FEB 1966. Other requests shall be referred to Air Force Materials Laboratory, Attn: RTD, Wright-Patterson AFB, OH 45433. This document contains export-controlled technical data.**
AUTHORITY
AFML ltr, 7 Dec 1972

AFML-TR-66-8

AD-479698

FRICTION AND WEAR BEHAVIOR OF SOLID FILMS

Paul J. Bryant and Paul L. Gutshall
Midwest Research Institute

AF 33(657)-10122

Technical Report AFML-TR-66-8
February 1966

479698

This document is subject to special export controls and each transmittal to foreign government or foreign nationals may be made only with prior approval of the Fluid and Lubricant Materials Branch, MANL, Nonmetallic Materials Division, Air Force Materials Laboratory Wright-Patterson Air Force Base, Ohio 45433

Air Force Materials Laboratory
Research and Technology Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

NOTICES

When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Copies of this report should not be returned to the Research and Technology Division unless return is required by security considerations, contractual obligations, or notice on a specific document.

AFML-TR-66-8

FRICTION AND WEAR BEHAVIOR OF SOLID FILMS

Paul J. Bryant and Paul L. Gutshall
Midwest Research Institute

FOREWORD

The subject of this research program is the friction and wear behavior of lamellar solids with emphasis on graphite. Dr. Tung Liu is the project engineer and monitor. The program is sponsored by the Air Force Materials Laboratory of the Research and Technology Division, Wright-Patterson Air Force Base, Ohio 45433. This report covers the period from 31 December 1964 to 31 December 1965. The manuscript was released by the author in December 1965 for publication as an RTD Technical Documentary Report.

The subject program was conducted in the Physics Section of Midwest Research Institute, 425 Volker Boulevard, Kansas City, Missouri 64110, under the direction of Dr. Sheldon L. Levy and Mr. Gordon E. Gross. The project leader is Dr. Paul J. Bryant. Research activities have been conducted by Dr. Paul J. Bryant, Mr. Paul L. Gutshall and Mr. Sidney A. Hamilton.

This technical report has been reviewed and is approved.

R. L. Adamczak

R. L. ADAMCZAK, Chief
Fluid & Lubricant Materials Branch
Nonmetallic Materials Division
AF Materials Laboratory

ABSTRACT

A new friction and wear system is described. The coefficient of friction and the wear rate of a single crystal or a compressed pellet sample may be measured in the same atmosphere as cleavage energy or stress-relaxation experiments are being conducted. This combined ability in one system gives a true basis for correlating data from fundamental measurements and actual frictional tests. The simultaneous experiments may be performed in ultra-high vacuum, or various controlled atmospheres.

Sets of data are reported for ~~the~~ friction and wear of graphite in two forms: pyrolytic graphite, and compressed graphite pellets. The two sample forms differ in both the size and relative orientation of their graphite lamellae. Data are compared for both sample forms in six environments: air, vacuum, oxygen, water, methanol and carbon tetrachloride.

~~The~~ effect of each environment was more pronounced on crystalline graphite samples than ~~on~~ pellet samples.

The most notable difference is the low wear rate which has been measured for pyrolytic samples in ultra-high vacuum. This absence of "dusting wear" accompanied by a moderate coefficient of friction for unannealed pyrolytic graphite is very interesting in regard to many possible applications.

An optical microscope study of ~~the~~ surfaces resulting from bulk shear of graphite crystals is presented. The majority of new area produced apparently results from unresolved lamellar shear. However, some of the applied shear stress appears to be resolved in cleavage and also dissipated into deformation of the sample. Similar experiments with molybdenite yield a truer shearing action; comparisons are described.

23p 9 ref 12 fig

(23 pp) (12 fig.) (0 tbls..) (9 ref.)

CONTENTS

ILLUSTRATIONS

I. INTRODUCTION

Lamellar solids had originally (Ref. 1) been described as intrinsic lubricants on the basis of their property of large interlayer spacing and the assumption of a complementary low shear strength. While this assumption is reasonable and does apply to many lamellar materials, there are exceptions and graphite is a notable one (Refs. 2 and 3).

Although graphite has been reported to fail as a lubricant in vacuum, it is very successful in air and under certain specific conditions. The high temperature integrity of graphite gives promise for many useful applications, if a workable set of conditions can be developed.

The objective of this research program includes both a basic understanding of the mechanisms by which graphite lubricates and predictions for practical applications of graphite. This report, together with previous reports (Ref. 4) and articles (Ref. 5), gives results for these objectives and the research efforts related to this program.

Initial results revealed some interesting characteristics for the basic graphite lattice. However, there was a question regarding the application of this fundamental knowledge to the actual frictional processes for graphite. A correlation of actual friction and wear data with the basic binding energy values has now given an over-all understanding of the friction mechanisms for graphite.

II. EXPERIMENTAL EQUIPMENT AND PROCEDURES

A special test chamber was designed and built for conducting friction and wear measurements on small graphite specimens. The test environment can be controlled from 10^{-9} torr to one atmosphere. Pure gases (O_2, H_2, Ar, N) and liquids (H_2O, CCL_4, CH_3OH) may be admitted into the system and the friction and wear measured. Crystalline graphite and compressed pellets were tested. Shear measurements were also made on Essex County graphite, using an Instron tensile testing machine. The following sections describe in detail the experimental equipment and techniques used in conducting these studies.

A. Ultra-High Vacuum Friction and Wear System

A UHV friction and wear system (Figure 1) was designed and constructed for the purpose of measuring the coefficient of friction and wear rates on small graphite samples. The testing environment may be accurately controlled and may be varied from 10^{-9} torr to one atmosphere of purified gas.

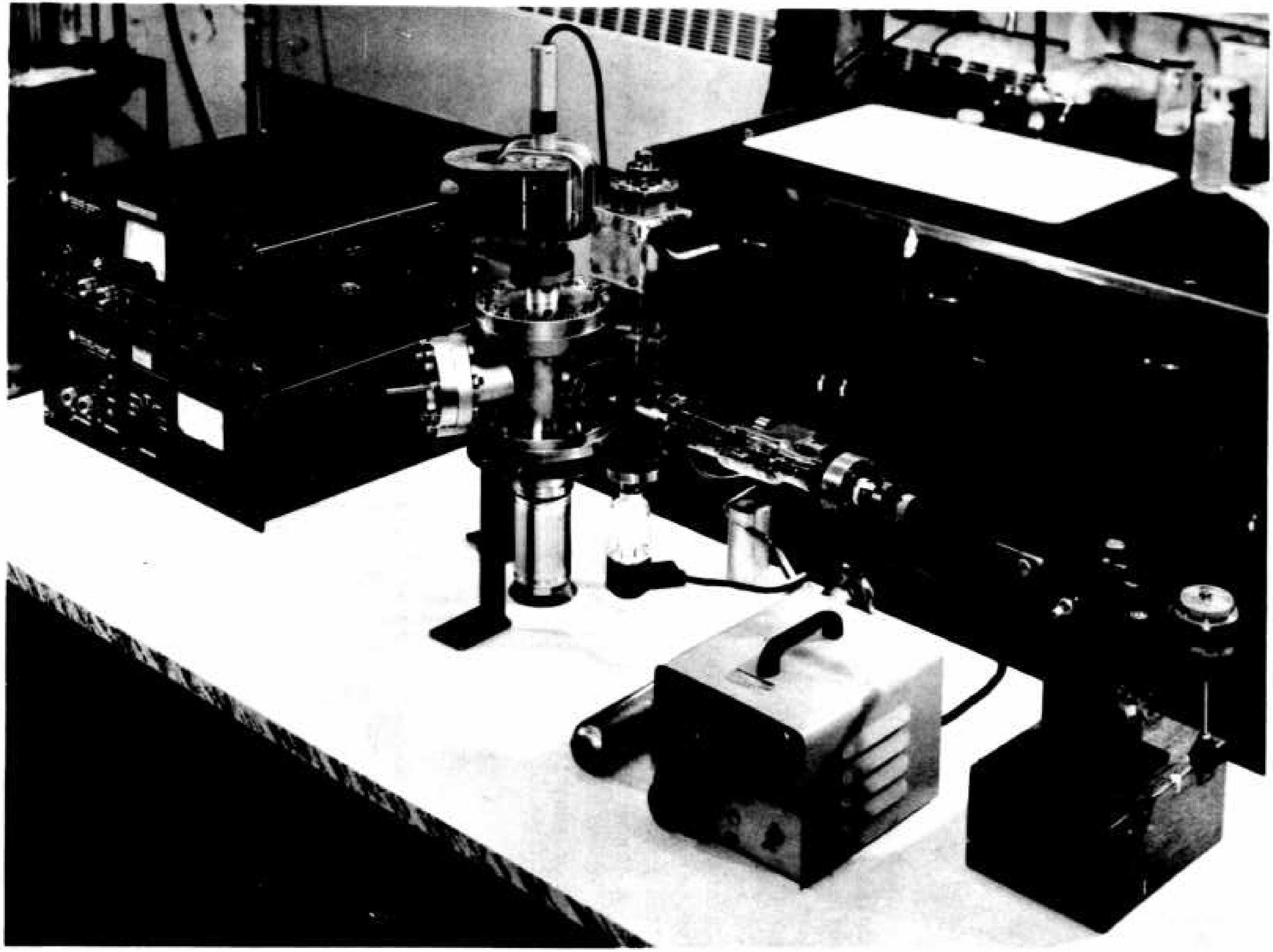

Figure 1 - Ultra-High Vacuum Chamber for Conducting Both Friction and Wear Experiments and Cleavage Experiments in the Same Environment. Cleavage chamber, right; manipulator shaft, left, for friction tests; rotary magnetic drive from below with variable speed motor drive, foreground; and getter-ion pump above.

1. Vacuum system: The vacuum chamber consists of a 4 in. diameter cylindrical shell with 6 in. diameter metal sealing flanges on both ends. An 8-liter-per-second getter-ion pump is used to maintain pressures of below 10^{-9} torr. The initial pump-out is accomplished through a roughing valve which is attached to the cross (Figure 2). The sample and wear track may be observed through a Pyrex view port.

2. Wear track assembly: A 4-in. stainless steel wear track assembly is mounted inside the UHV chamber. The wear track is supported from the 6-in. pump flange as shown in Figure 3. The track is mounted between two fixed plates (Figure 4) and lubricated with an MoS_2 dry film (MLF-5) (Ref. 6). The track is centered by a sleave bearing. Since the support for the track is directly above the sample, large normal loads may be applied to the sample without applying a torque to the track assembly.

A variable speed motor (0-3,000 rpm) is used to drive the track. A magnetic drive is used to connect the motor to the track assembly. A flexible coupling between the track and magnetic drive eliminates any vibrations coming from the motor. The wear track may be driven to linear speeds of 500 ft/min. All lubricating materials in the drive and track assembly are bakeable to 400°C for extended periods, thus allowing a complete bake-out of the sample and chamber before testing.

3. Coefficient of friction measurements: A special apparatus to record coefficients of friction is attached to the UHV chamber after bake-out has been accomplished (Figures 5 and 6).

The apparatus is used outside the vacuum system but attaches directly to a shaft which passes through a flexible metal bellows to the sample. A normal load is applied to the sample by attaching weights to this same shaft extension. The extension arm is rigid in the vertical direction, so as to effectively transmit the normal load. However, the arm is somewhat flexible in the horizontal direction so that some deflection will be caused by a frictional force (see Figure 6). Four sensitive strain gauges record this deflection and thus give a measurement of frictional force.

The four strain gauges, which are mounted on the shaft extension to the sample, are calibrated. They are arranged in a Wheatstone bridge circuit with two of the gauges as temperature compensators. The other two gauges are mounted on either side of the deflection shaft and connected so that their output signals add to raise the net sensitivity. A balance method was employed for calibration using known weights at the sample position to produce deflections equivalent to known friction force values. The frictional force sensitivity is equal to 1 gm/cm on the recording chart paper; thus, frictional force values of fractional grams can be detected.

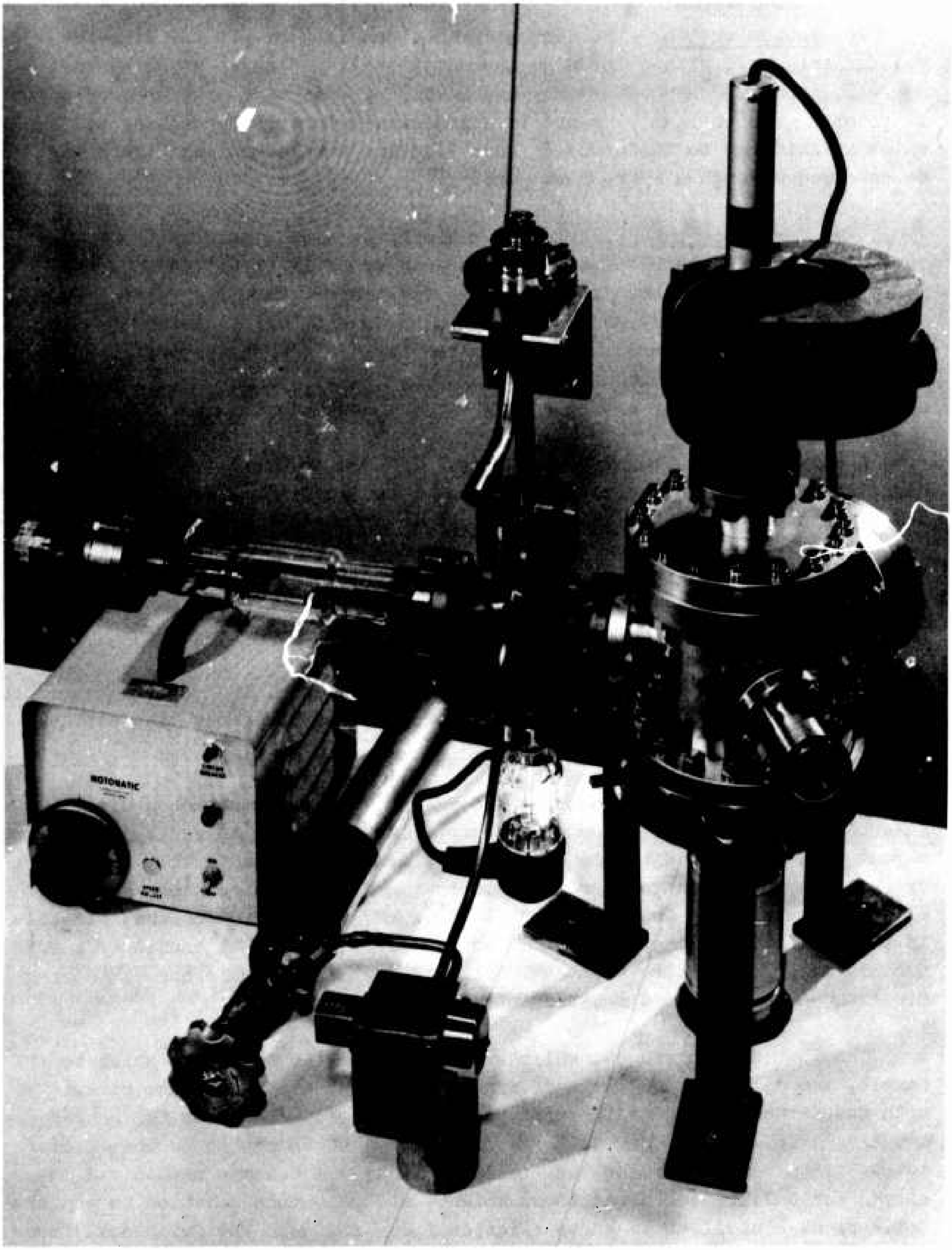

Figure 2 - Gases Can Be Admitted into the Chamber from a Research Grade Bottle Through a Variable Leak Valve. Controlled atmospheres from the ultra-high vacuum range to 1,000 torr may be provided.

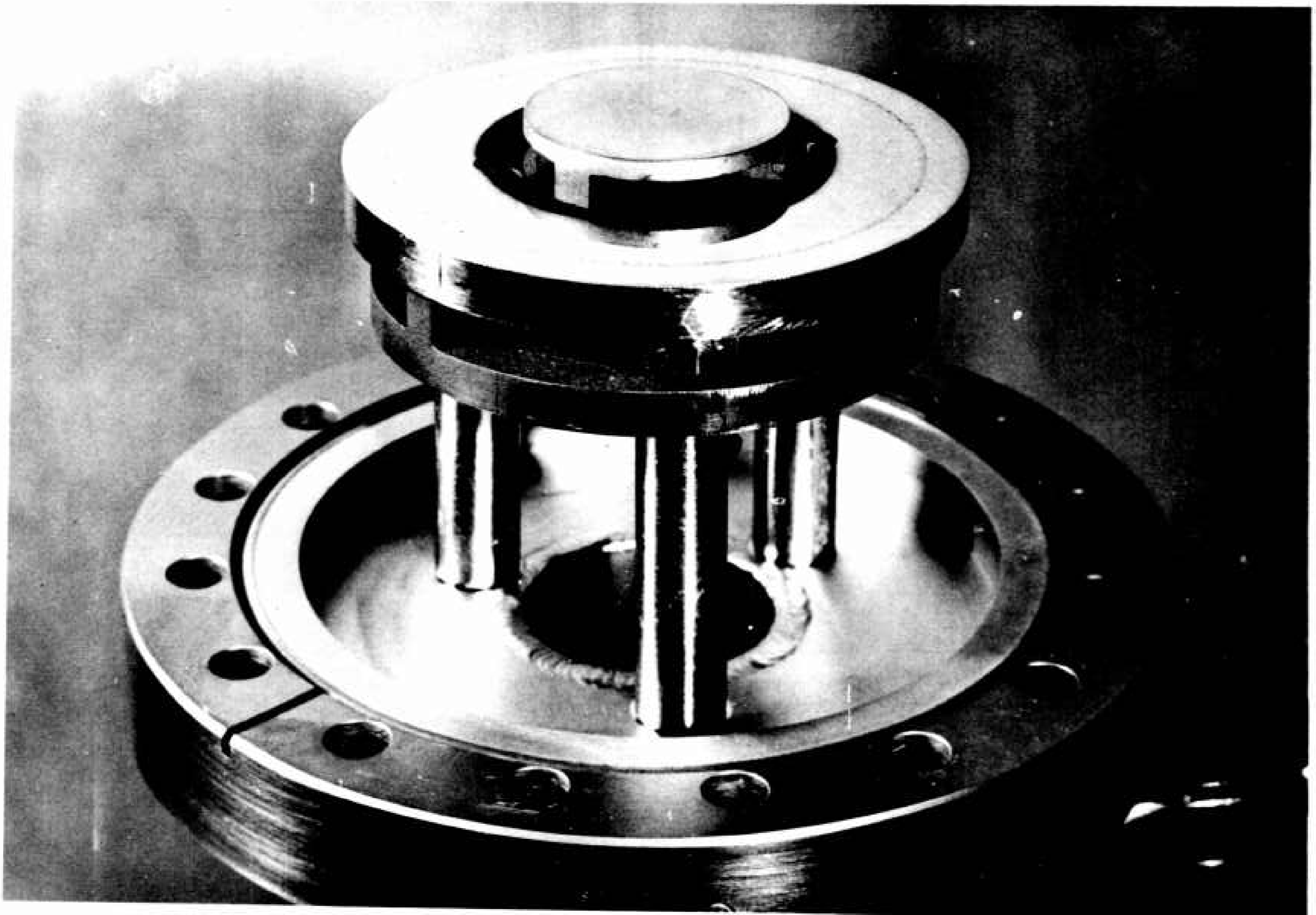

Figure 3 - Stainless Steel Wear Track is Supported Between Two Stationary Plates. A graphite wear track can be seen on the surface of the metal track.

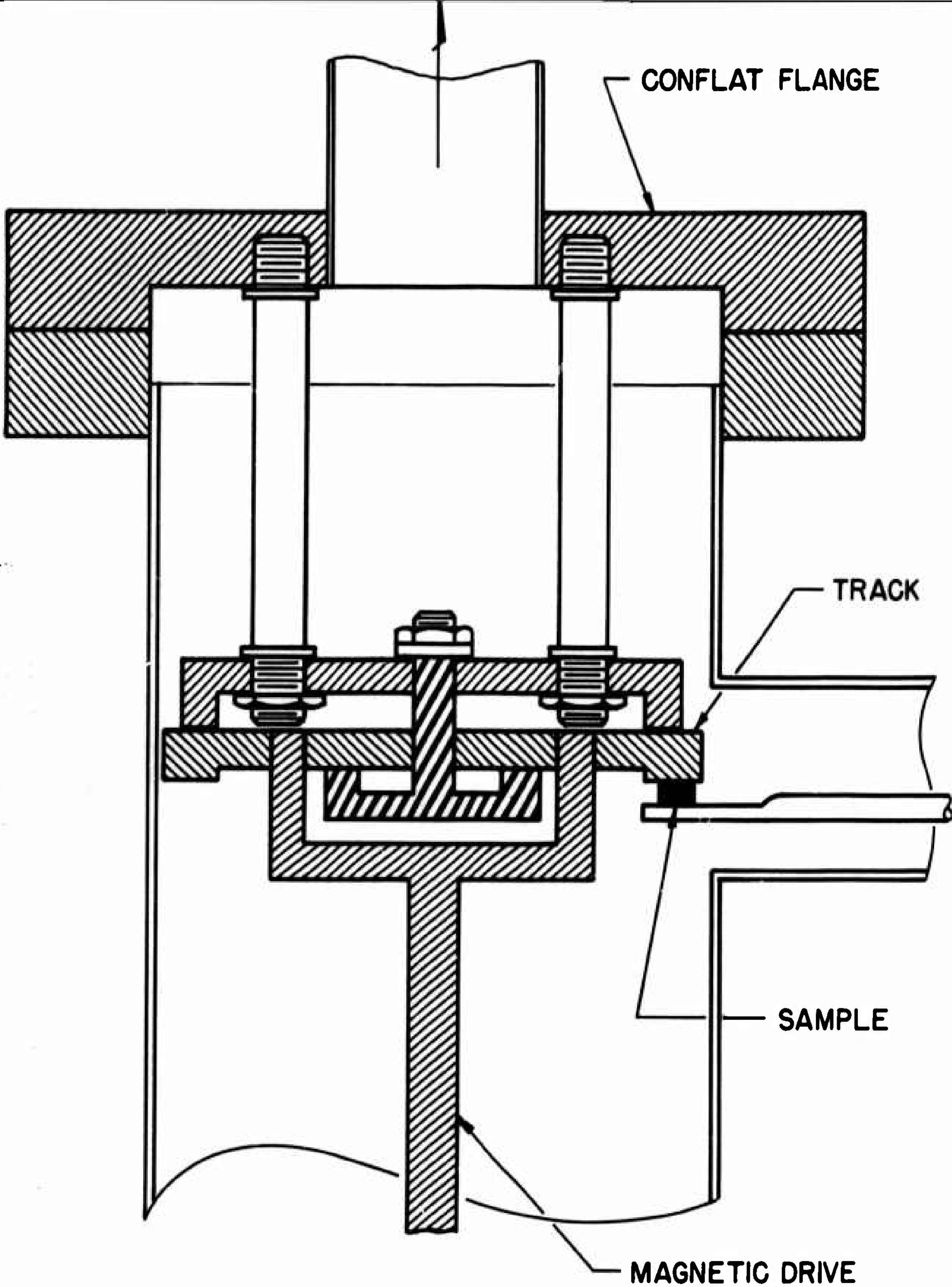

Figure 4 - Cross Section View of the Friction and Wear Chamber. The wear track support is lubricated with a MoS_2 film composition (MLF-5).

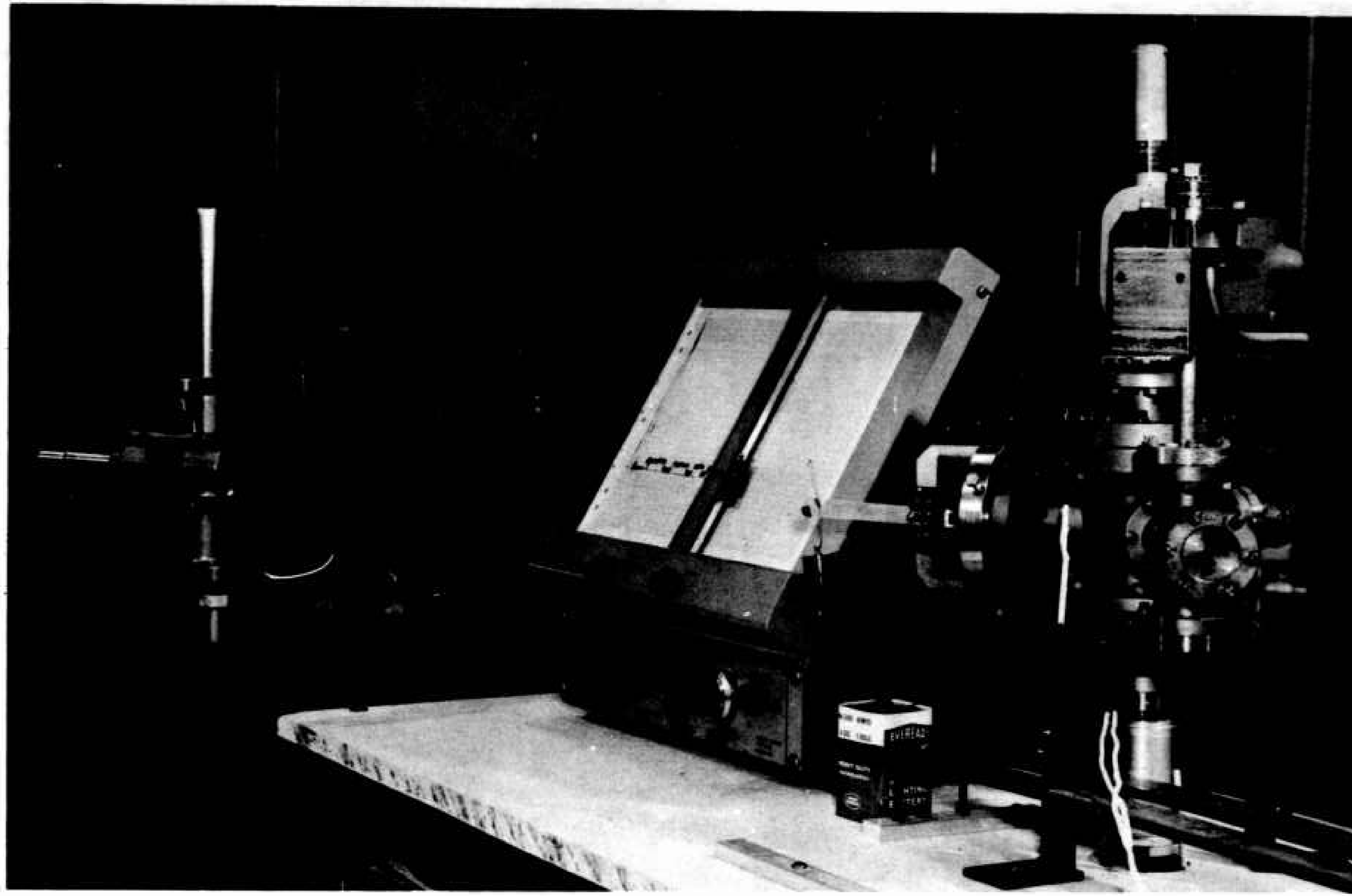

Figure 5 - Ultra-High Vacuum Chamber for Conducting Friction and Wear Experiments in Accurately Controlled Environments. Frictional data are measured with strain gauges and wear is measured using a cathetometer.

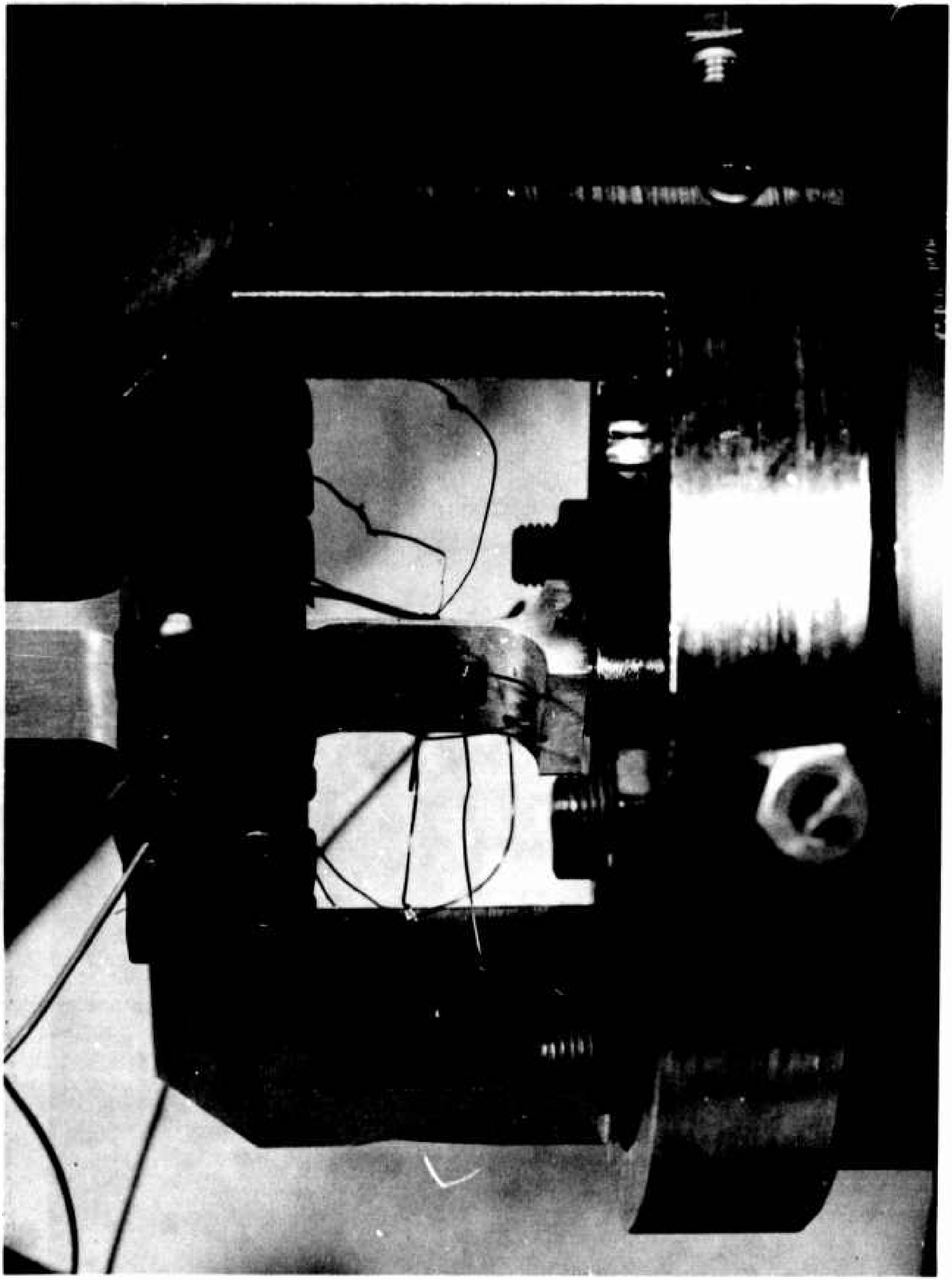

Figure 6 - Friction Force Measuring Device Which is Attached to the Shaft Holding a Specimen. Strain gauges which are mounted on a thin section of the shaft measure the frictional force.

4. Wear rate measurements: Wear rate measurements were made by monitoring the displacement of the sample holder (extension arm of the friction measuring apparatus). Two methods were used in making these measurements. (1) A cathetometer was used to make periodic measurements and to measure total wear during the test. (2) An optical displacement sensor (Photopot)* was used to continuously measure the wear rates during the friction test. This sensor was mounted on the end of the extension arm and the printout signal was recorded as a function of time during the test.

B. Environmental Control

Since one of the main purposes of this study was to measure the effect of various gases on the frictional properties of graphite, methods of introducing pure gases were developed.

Pure gases (O_2, Ar, H_2, CO_2) were admitted through a UHV leak valve from a research grade gas bottle. The line connecting the gas bottle to the vacuum system was baked at 400°C to remove any impurity.

The admission of various pure liquids (CCl_4, H_2O, CH_3OH) was required for a number of experiments. A technique involving an evacuated glass capillary which could be inserted into a sealed ampoule and then broken within the liquid was reported earlier (Ref. 7). The above technique used a ground glass valve to limit the amount of liquid admitted to the finger while a second variable leak valve was used to regulate the flow into the friction and wear chamber itself. The ground glass valve was eliminated by employing a small piece of pliable material in the bottom of the ampoule. Contact of this pliable pad against the broken capillary serves as a valve to limit the amount of liquid entering the finger.

C. Preparation and Mounting Friction and Wear Specimens

Two basic types of graphite were used in the friction and wear experiments. Crystalline (unannealed pyrolytic) graphite samples were prepared from the same material previously used for studying the cleavage energy in vacuum and various atmospheres. Compressed pellet samples were prepared from Dixon Brand flake graphite.

Crystalline graphite samples were cut in the form of parallelepipeds, 5 mm. x 5 mm. x 2 mm. The specimens were mounted on the sample rod by stainless steel clamps. In all the tests conducted, the orientation of the sample was such that the basal plane was parallel to the plane of the track.

* Giannini Controls Corp., Duarte, California.

Compressed pellets, 1/4 in. diameter x 3/16 in. long, were pressed from extra fine powdered flake material. The pellets were dry formed under 37,000 psi. The compressed pellets were also mounted by stainless steel clamps.

D. Shear Stress Measurements on Essex County Graphite Single Crystals

Essex County graphite single crystals were extracted from their marble matrix and the shear strength was measured in air. The single crystals were removed from the marble by etching hydrochloric acid. The crystals were rinsed in distilled water and then stored in a dry box for several days.

The shear strength of the graphite single crystals was measured in an Instron tensile testing machine. One side of a graphite single crystal was cemented to a special clamp with Eastman 910 cement. The clamp and specimen were then mounted in the Instron machine. A second clamp was then brought into contact with the other side of the sample and cemented into place. Shear stresses could then be measured using standard tensile testing techniques.

III. RESULTS AND DISCUSSION

Cleavage energy values and stress-relaxation data for the materials tested have been recorded for various atmospheres of interest to the lubrication process. Shear strength measurements have also been recorded. The high value of intrinsic binding energy as observed in vacuum together with the relatively high shear strength has provided an understanding of the basic lack of lubricating ability. In addition, the lower cleavage energy and stress-relaxation data for graphite in polar and chemically active atmospheres has provided an understanding of the lubrication mechanism. The experiments described below correlate this fundamental knowledge with friction and wear data.

A series of friction and wear tests has now been completed for graphite pellets and crystalline samples in a number of relevant environments. The conditions for these tests have included all of those for which cleavage data were obtained so that a correlation of basic properties such as binding energy values, and stress-etch phenomena may be applied to the results of these practical frictional tests.

A. Frictional Data for Graphite in Various Environments

The coefficient of sliding friction was measured for crystalline pyrolytic graphite and for graphite pellets sliding on a graphite wear track in the presence of relevant atmospheres. A linear sliding velocity of 240 cm/sec and a 50-gm. load were used throughout. Each result is truly representative for the individual environmental condition since each test was completely run in that one environment. That is, the test system was degassed at high temperature to establish a noncontaminating environment, then the pellet and wear track were stabilized in the dusting wear condition; finally, the pellet and track combination was established under the influence of only the one pure gas to be tested. Thus, residual environments or conditions, such as a previously established wear track, were eliminated so that all parameters of the individual tests are representative of the specific environment under study.

1. Frictional data for graphite crystallites in pellet form: The environmental effects upon the frictional properties of graphite pellets were examined for six environments: vacuum, air, water vapor, oxygen, methanol and carbon tetrachloride. The test pressures were varied from about 10^{-7} torr up to the vapor pressure of each material at room temperature. For example, pure methanol was admitted to the UHV test chamber until the gas and liquid phases reached equilibrium for room temperature (100 torr).

Figures 7 through 10 show the coefficients of friction of graphite pellets operated in: oxygen, water, methanol and carbon tetrachloride, respectively. Since these plots give the directly recorded data from individual experiments, there are some variations in the starting values. Note, that each gas, except oxygen, has a pronounced effect upon the frictional value near the same pressure level of 10^{-1} torr. This rather uniform pressure behavior indicates that the same mechanism may be operative in each case for H_2O, CH_3OH, and CCl_4. The rather abrupt change in the coefficient of friction suggests the adsorbed film mechanism.

Since the boiling point values for these three materials are all close to the same level (65, 77 and 100°C), the condensation of an adsorbed film should occur near the same pressure value as observed.

Oxygen on the other hand has a boiling point which is much lower than the three materials mentioned above. Therefore, the adsorbed film effect should not occur until a much higher partial pressure of oxygen is reached. However, Figure 7 shows that a reduction of friction due to O_2 occurs for about one-half the pressure value required for the other three materials. This contradiction indicates that oxygen affects graphite lubrication by a different mechanism as discussed below.

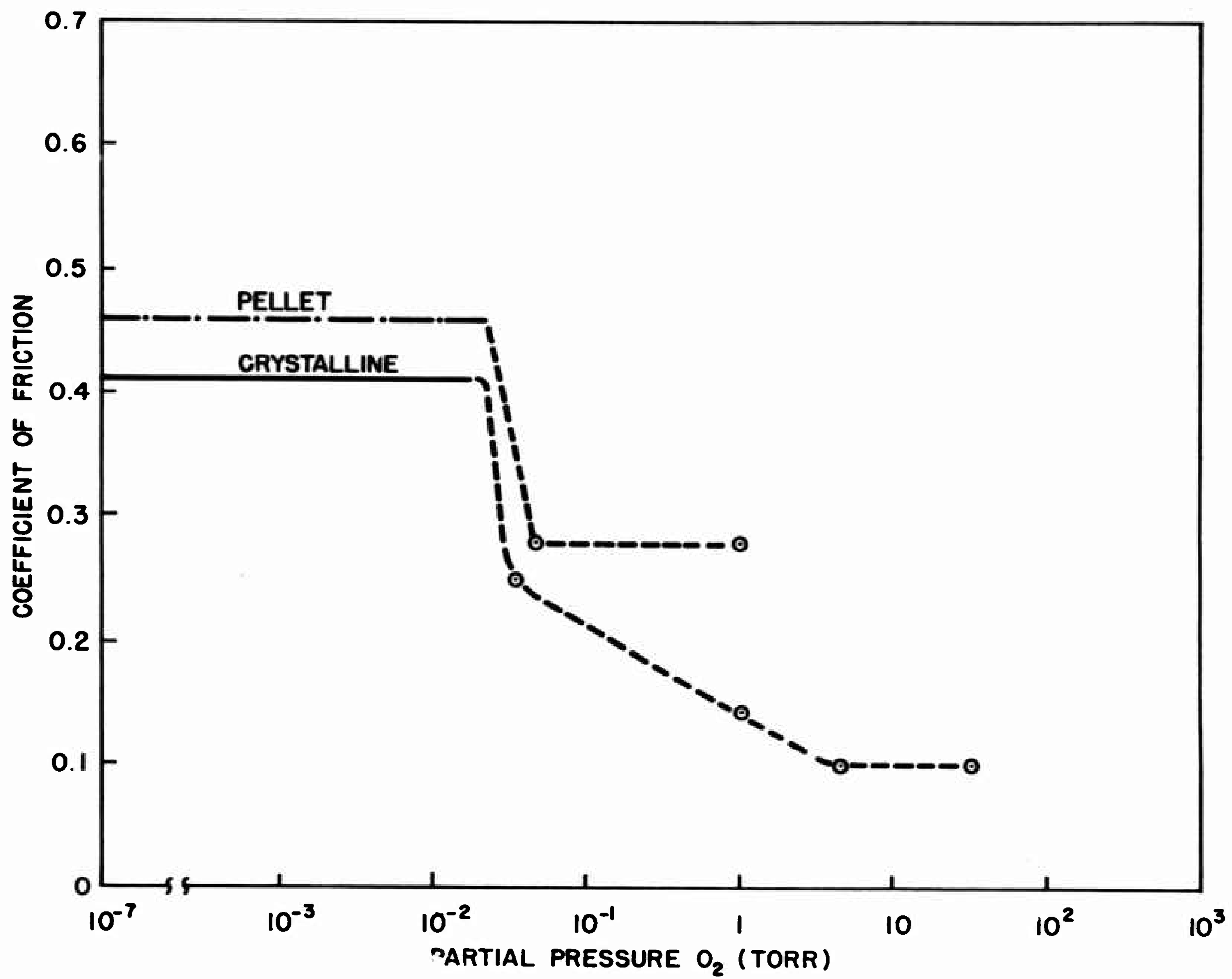

Figure 7 - The Coefficient of Sliding Friction as a Function of the Partial Pressure of Oxygen

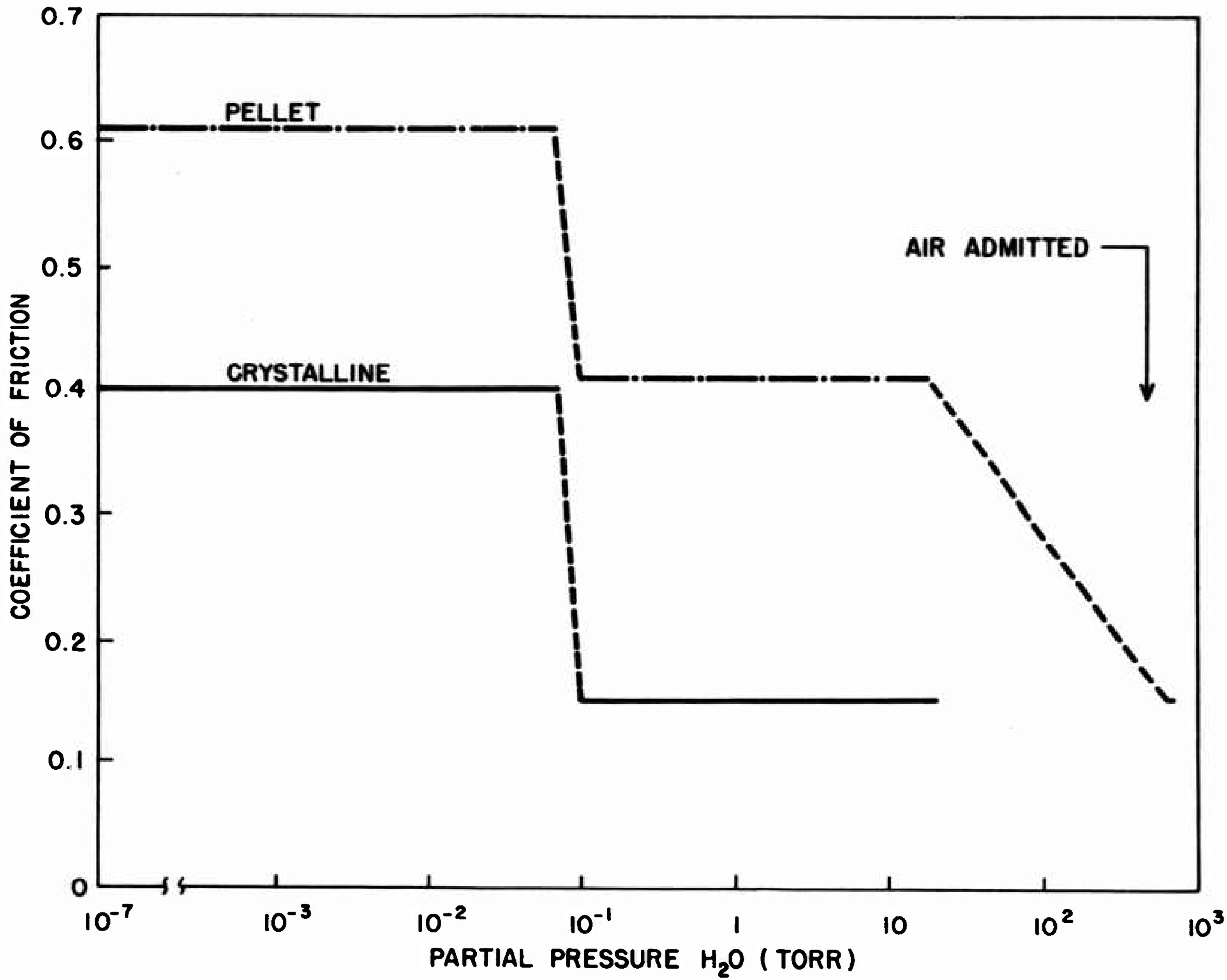

Figure 8 - The Coefficient of Sliding Friction as a Function of the Partial Pressure of Water Vapor

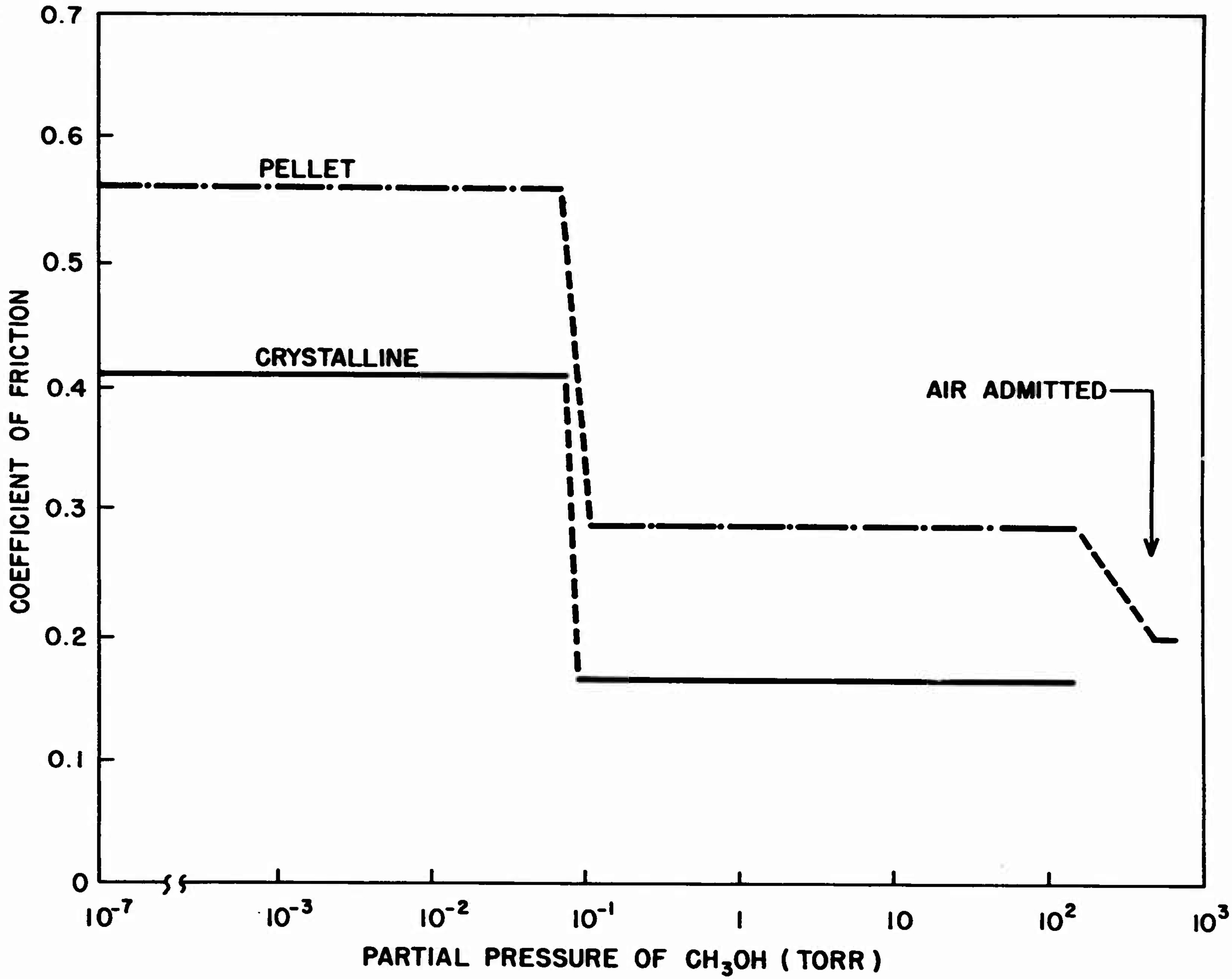

Figure 9 - The Coefficient of Sliding Friction as a Function of the Partial Pressure of Methanol

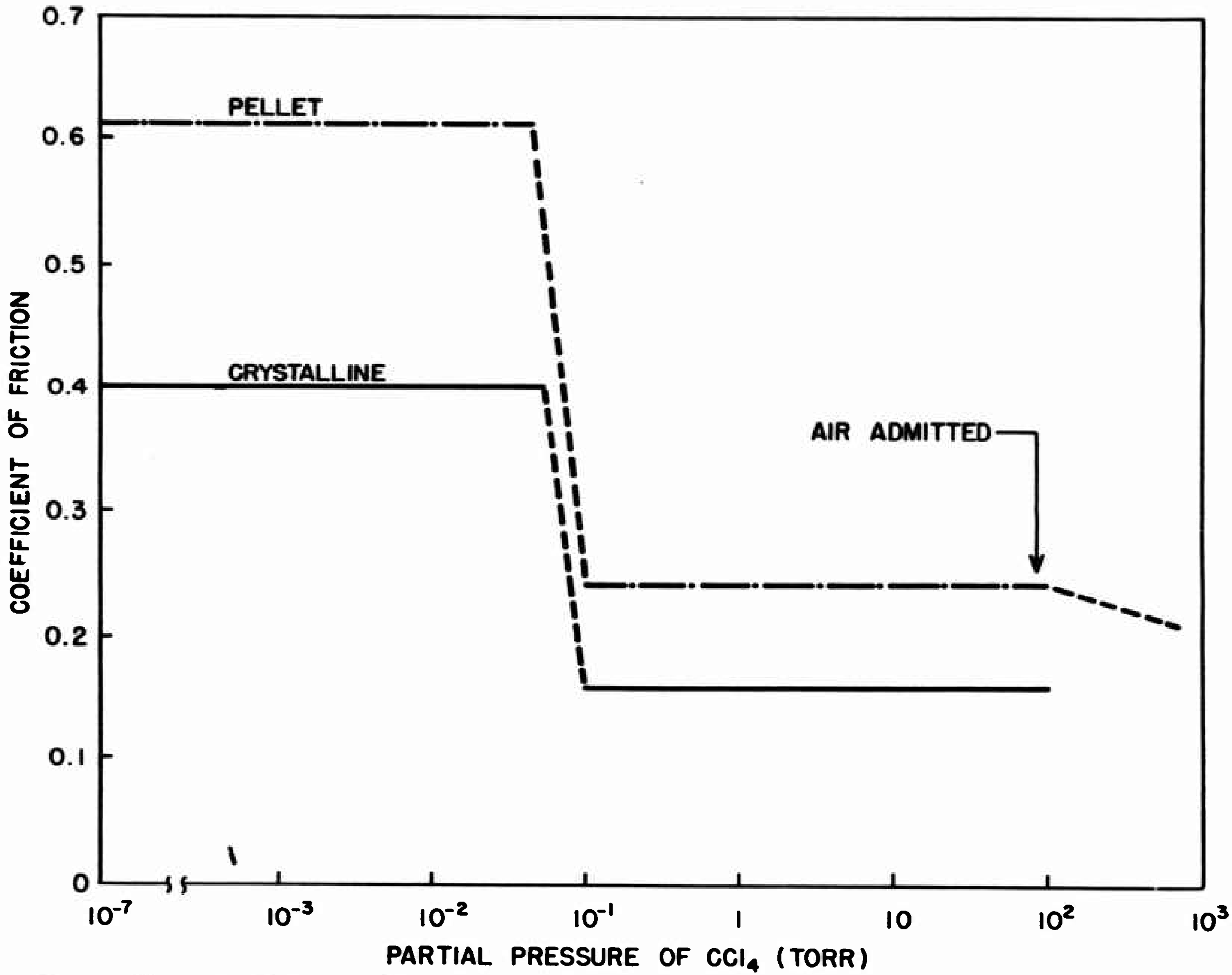

Figure 10 - The Coefficient of Sliding Friction as a Function of the Partial Pressure of Carbon Tetrachlori

Cleavage experiments with graphite have shown that oxygen has an intrinsic effect upon the graphite lattice. The stress-etch process described (Ref. 5) for oxygen holds the greatest potential for reducing the interlamellar binding energy of graphite. The strong chemical reaction between the π-electrons of graphite and the unpaired electrons of oxygen explains the effect observed at an oxygen pressure below that expected for physical adsorption. Thus, the results of friction tests with graphite pellets are in agreement with the expected behavior for graphite.

The coefficient of friction for graphite pellets in vacuum shows some variation for the individual data plots given in Figures 7 through 10 and the average value is rather high. An explanation for these high values may be derived from the test procedure which required the establishment of high wear and the destruction of any previous wear track. That is, an equilibrium condition was established in an ultra-high vacuum environment before the data were listed as representative for the friction of graphite in vacuum. Such an equilibrium frictional condition was established in vacuum prior to every test for each of the known environments. Coefficient of friction values often began at the 0.4 level following bake-out of the system, but rose to the 0.6 level after a 5- to 20-min. running time in vacuum.

2. <u>Frictional data for crystalline pyrolytic graphite</u>: Crystalline graphite samples in both the naturally deposited form from Essex County, New York, and in the form as grown by the pyrolysis method were tested. The Essex County crystals were found to be the largest and most perfect natural graphite samples from a list of sources examined: Alabama flake, Ceylon, Pacific, and Madagascar. A number of relatively large Essex County crystals were removed from the original granite matrix and mounted for shear measurements and friction tests. Results from the shear experiments are given in Section C below. The friction tests were hampered by the thin dimensions of the crystal flakes and the need for bonding to a holder. Results were questionable due to the probable contact of binder and track in addition to the crystal contacts with the track. Since these tests and cleavage experiments gave similar results for both natural and pyrolytic graphite, it was decided to use the pyrolytic samples for the bulk of the testing program.

The most outstanding result obtained with pyrolytic graphite was the very low wear as described in Section B below. The frictional properties discussed here also show a significant difference from those of pellets. In each case, Figures 7 through 10, the coefficient of friction is noticeably lower for the crystalline samples than for pellets. This is uniformly true for unannealed pyrolytic graphite operated in vacuum or in the four environments: O_2, H_2O, CH_3OH and CCl_4. In air the coefficients become essentially equal; the pellets record a coefficient of 0.10 and crystalline samples operate slightly lower at 0.08.

The coefficient of friction for unannealed pyrolytic graphite running in vacuum is rather moderate and very uniform (0.40 to 0.41) as seen in Figures 7 through 10. In each of the tests with pyrolytic samples the graphite lamellae were carefully aligned parallel to the wear track.

Annealed pyrolytic graphite was also examined and found to be relatively impractical for direct application. The material is so soft that clamping arrangements such as those used with pellets are not practical. Various bonding cements were tried to hold the samples onto a flat plate. However, under friction testing the entire annealed sample would separate from the holder leaving only a thin surface layer with the glue bond. Even the special glass cement with annealing treatment could not prevent this problem. Of course, the separation of the main sample from a surface layer is due to the properties of the sample rather than the cement. A successful mount was finally achieved by shaping a depression in a metal holder to accept the sample with a close fit so that the cement could bond all edges of sample.

Results obtained with the annealed pyrolytic graphite gave some interesting indications. The coefficient of friction was very high in vacuum ($\mu = 0.76$), but the wear was very low. The high friction value may have been due to the fact that no track was established by this soft, highly oriented crystalline material. An analogy to this situation has occurred before with single crystal lamellar samples. There was also an indication that the coefficient of friction for the annealed samples is 0.4, the same as for unannealed, when running on an established wear track in vacuum.

B. The Wear of Graphite Sliding in Various Environments

The wear rate of graphite is probably the most important property relating to application as a vacuum lubrication. Even the moderately high coefficient of friction (0.41) for pyrolytic graphite in vacuum can be tolerated if the wear rate is low enough to allow useful lifetimes. The very interesting result obtained from the present tests is the negligible wear rate observed for pyrolytic graphite under a 50-gm. load even in ultra-high vacuum. A 50-gm. load was also used for the pellet tests and the high dusting wear which is characteristic of graphite compacts was measured.

The wear rate of graphite compacts was measured for sliding at 240 cm/sec in six different environments: air, vacuum, O_2, H_2O, CH_3OH, and CCl_4. A sensitive photopot arrangement was used to register the wear versus time on a chart recorder.

The wear rate in vacuum was very high and could be described appropriately by the term, dusting wear. For the linear sliding velocity of 240 cm/sec and a 50-gm. load the wear rate in vacuum was 0.006 mm/min.

A negligible wear rate was recorded for graphite pellets operating in each of the environments, O_2, H_2O, CH_3OH and CCl_4, when the partial pressure was about 1 torr or higher. Minimum pressure values of H_2O, CH_3OH and CCl_4 for reduction of wear rates agreed very closely with those published by Savage (Ref. 8) with values in the range from 10^{-2} torr to 1 torr.

Data for the minimum partial pressure of oxygen were very interesting since they fulfilled the predictions from earlier binding energy measurements. As discussed earlier, oxygen would be expected to attack the π-electron bonds of graphite at a significant rate to cause friction and wear reduction from the gas phase without the need of an adsorbed film. Due to the low boiling point of oxygen, an adsorbed film would not be expected at room temperature for pressures around 10^{-1} torr. The reductions of friction and wear observed for pressures near 10^{-1} torr of oxygen are apparently due to the strong oxygen attack upon graphite bonding (Ref. 5).

C. Examination of Surfaces Resulting from Shear of Graphite Single Crystals

Single crystal samples of Essex County graphite were mounted in an Instron tensile testing machine in air and a shear stress was applied parallel to the basal plane. The samples yielded plastically before failure which indicated that basal slip did occur. Examination of the samples after testing revealed that cleavage had also occurred. The relative amount of the two types of failure may possibly be inferred from the optical micrographs (see Figures 11 and 12). A clean lamellar separation occurred over the majority of the sample area seen in Figure 11. Several areas of partially cleaved material are clearly visible in the form of folded or rolled back lamellae. One thin partially cleaved lamellae is visible in Figure 11 with a portion so thin that it is optically transparent. Interference patterns develop under transmitted lights, as seen in Figure 11. This occurrence also confirms the indicated cleavage, i.e., thin folded back portions are identifiable by this means. In addition, other areas show the remnants of thicker cleavage layers. Therefore, we may conclude from this optical microscopy study that at least a portion of the shearing motion is resolved into a cleavage action.

The majority of new surface, which was created from the bulk of the crystal by the shearing motion, shows smooth lamellar separation. Whether this smooth separation resulted from a pure shearing action or from a quasi-cleaving action is not readily discernible. Two new surfaces are created by separating into the bulk of the crystal. Both have been examined and found to be essentially the same in regard to the per cent of partially cleaved debris. That is, the amount of cleaved material visible on the surface of Figure 11 is representative.

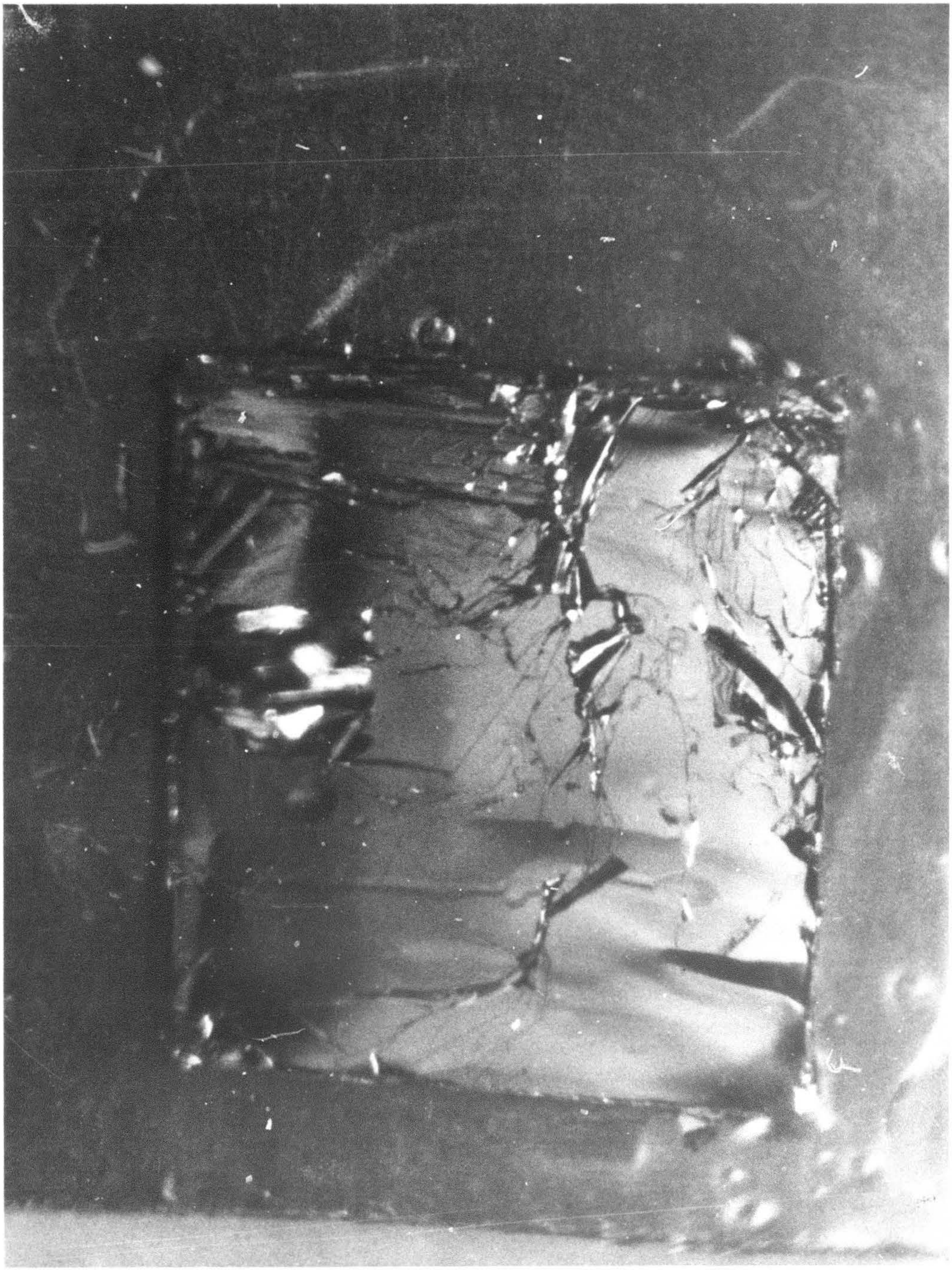

Figure 11 - Optical Micrograph of a Sheared Surface of a Graphite Single Crystal. Evidence of both shear and partial cleavage are present on the surface.

Figure 12 - Optical Micrograph of a Graphite Single Crystal Which was Sheared Parallel to the Basal Plane with More Induced Deformation Than Seen in Figure 11

Figure 12 shows a surface produced from the bulk of another graphite single crystal. The two views, Figures 11 and 12, show a variation in the amount of sample wrinkling or distortion for various samples with a higher degree in Figure 11. Since the distortion seen in Figure 11 is oriented preferentially to the direction of the shearing action, it is quite possible that the distortion was induced by this action. The amount of distortion observed on a molybdenite surface produced by the same bulk shearing action is less than observed for graphite. The molybdenite surface is noticeably smoother than even the better graphite surface seen in Figure 11. It may therefore be meaningful to correlate the six times higher shear strength measure (Ref. 9) for graphite, in relation to molybdenite, with the greater amount of distortion observed on graphite samples following the shear strength test. That is, the high value of interlayer binding energy for graphite may not only be exhibited in terms of cleavage measurements, stress-relaxation tests, and shear strength values, but also as a distortion of the crystals during shear tests.

Although these graphite crystals were sheared in air, it is unlikely that an interaction of air could occur to influence the bulk shear or internal bulk cleavage processes. Thus, the six times higher shear strength for graphite over molybdenite and the greater distortion caused in the graphite crystals correlates with the intrinsic lubrication nature of molybdenite versus graphite which is not an intrinsic lubricant.

IV. CONCLUSIONS

A number of interesting conclusions can be drawn from the results of these applied friction tests. Some of the results were predicted by the cleavage experiments while others were completely new.

Previous results (Ref. 5) have shown that a basic property of graphite is significantly affected by the interaction of oxygen, and that property is the interlamellar binding energy as measured by cleavage experiments. An important question has been raised regarding the relevance of such measurements to actual lubrication phenomena. This question bears particular significance to the oxygen experiment, since other reliable results (Ref. 3) had not shown an equivalent oxygen effect for a partial pressure below 200 torr, i.e., below atmospheric content. The apparent difference between these results was thought to be due to the use of crystalline graphite samples for cleavage measurements, whereas carbon brush material was used for the lubrication experiments. Results reported here remove the apparent conflict by showing that the oxygen effect upon graphite compressed pellet samples is not large. In addition, the present results have shown that a strong oxygen effect exists for highly oriented crystalline graphite in frictional

testing as predicted from the cleavage measurements in oxygen. All of these latter results are for oxygen partial pressures below the atmospheric content.

The effect of pure oxygen alone upon the lubrication ability of compressed pellets was so small that it could be easily overlooked or considered to be nonexistent. However, the effect of oxygen alone upon the coefficient of friction of crystalline pyrolytic graphite was as great as the effect caused by air. A large reduction of the coefficient of friction was anticipated from the mechanism of oxygen attack upon the binding energy of graphite (Ref. 5).

Friction and wear tests with crystalline graphite samples have now shown agreement with the mechanisms proposed (Ref. 1) from cleavage experiments in various environments. In particular, the effect of oxygen, which was determined from cleavage and strain-relaxation experiments, has been confirmed from actual frictional tests. Other results also show correlation between coefficient of friction data and cleavage energy measurements, so that the binding energy criterion is now well established and applicable to practical conditions.

REFERENCES

1. Bragg, W. L., Introduction to Crystal Analysis, G. Bell and Son, London, 64 (1928).

2. Ramadanoff, D., and S. W. Glass, "High Altitude Brush Problem," Elec. Eng., 63, 825 (1944).

3. Savage, R. H., "Carbon-Brush Contact Films," Gen. Elec. Rev., 48, 13 (1945); "Graphite Lubrication," Jour. Appl. Phys., 19, 1 (1948).

4. Bryant, P. J., WADD Technical Report 60-529, Part I, USAF (1960).

   Bryant, P. J., WADD Technical Report 60-529, Part II, USAF (1961).

   Bryant, P. J., Technical Documentary Report RTD-TDR-63-4291 (1964).

   Bryant, P. J., Technical Documentary Report AFML-TR-65-5 (1965).

5. Bryant, P. J., P. L. Gutshall, and L. H. Taylor, "A Study of Mechanisms of Graphite Friction and Wear," WEAR, 7, 1, 118 (1964).

   Bryant, P. J., L. H. Taylor, and P. L. Gutshall, "Cleavage Studies of Lamellar Solids in Various Gas Environments," 1963 Transactions of the Tenth National Vacuum Symposium, 21 (1963).

   Bryant, P. J., "Cohesion of Clean Surfaces and the Effect of Adsorbed Gases," 1962 Transactions of the Ninth National Vacuum Symposium, MacMillan Press, Washington, 311 (1963).

   Bryant, P. J., and C. M. Gosselin, "UHV Controlled Atmosphere Test Chamber Using Mercury Vapor Pump Sets," 1961 Transactions of the Eighth National Vacuum Symposium, 1224 (1962).

6. Hopkins, V., D. H. Gaddis, and R. D. Hubbell, Proc. USAF Aerospace Bearing Conference, 80 (1964).

7. Bryant, P. J., Technical Documentary Report, AFML-TR-65-5, p. 6 (1965).

8. Savage, R. H., and D. L. Schaeffer, Jour. Appl. Phys., 17, 136 (1956).

9. Bryant, P. J., Technical Documentary Report, AFML-TR-65-5 (1965).

Security Classification

DOCUMENT CONTROL DATA - R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Midwest Research Institute 425 Volker Boulevard Kansas City, Missouri 64110	2a. REPORT SECURITY CLASSIFICATION Unclassified 2b. GROUP

3. REPORT TITLE

FRICTION AND WEAR BEHAVIOR OF SOLID FILMS

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Annual Report (31 December 1964 to 31 December 1965)

5. AUTHOR(S) (Last name, first name, initial)

Bryant, Paul J.
Gutshall, Paul L.

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
February 1966	23	9

8a. CONTRACT OR GRANT NO. AF 33(657)-10122 b. Project No. 7342 c. Task No. 734204 d.	9a. ORIGINATOR'S REPORT NUMBER(S) AFML-TR-66-8 9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)

10. AVAILABILITY/LIMITATION NOTICES This document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of the Fluid and Lubricant Materials Branch, MANL, Nonmetallic Materials Division, Air Force Materials Laboratory, Wright-Patterson AFB, Ohio.

11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY
	Air Force Materials Laboratory (RTD) Air Force Systems Command Wright-Patterson AFB, Ohio 45433

13. ABSTRACT

A new friction and wear system is described. The coefficient of friction and the wear rate of a single crystal or a compressed pellet sample may be measured in the same atmosphere as cleavage energy or stress-relaxation experiments are being conducted. This combined ability in one system gives a true basis for correlating data from fundamental measurements and actual frictional tests. The simultaneous experiments may be performed in ultra-high vacuum, or various controlled atmospheres. (U)

Sets of data are reported for the friction and wear of graphite in two forms: pyrolytic graphite, and compressed graphite pellets. The two sample forms differ in both the size and relative orientation of their graphite lamellae. Data are compared for both sample forms in six environments: air, vacuum, oxygen, water, methanol and carbon tetrachloride. (U)

The effect of each environment was more pronounced on crystalline graphite samples than on pellet samples. (U)

(Continued)

14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Solid Lubricant Friction and Wear in Vacuum Environmental Effects on Friction and Wear Pyrolytic Graphite						

INSTRUCTIONS

1. ORIGINATING ACTIVITY: Enter the name and address of the contractor, subcontractor, grantee, Department of Defense activity or other organization *(corporate author)* issuing the report.

2a. REPORT SECURITY CLASSIFICATION: Enter the overall security classification of the report. Indicate whether "Restricted Data" is included. Marking is to be in accordance with appropriate security regulations.

2b. GROUP: Automatic downgrading is specified in DoD Directive 5200.10 and Armed Forces Industrial Manual. Enter the group number. Also, when applicable, show that optional markings have been used for Group 3 and Group 4 as authorized.

3. REPORT TITLE: Enter the complete report title in all capital letters. Titles in all cases should be unclassified. If a meaningful title cannot be selected without classification, show title classification in all capitals in parenthesis immediately following the title.

4. DESCRIPTIVE NOTES: If appropriate, enter the type of report, e.g., interim, progress, summary, annual, or final. Give the inclusive dates when a specific reporting period is covered.

5. AUTHOR(S): Enter the name(s) of author(s) as shown on or in the report. Enter last name, first name, middle initial. If military, show rank and branch of service. The name of the principal author is an absolute minimum requirement.

6. REPORT DATE: Enter the date of the report as day, month, year; or month, year. If more than one date appears on the report, use date of publication.

7a. TOTAL NUMBER OF PAGES: The total page count should follow normal pagination procedures, i.e., enter the number of pages containing information.

7b. NUMBER OF REFERENCES: Enter the total number of references cited in the report.

8a. CONTRACT OR GRANT NUMBER: If appropriate, enter the applicable number of the contract or grant under which the report was written.

8b, 8c, & 8d. PROJECT NUMBER: Enter the appropriate military department identification, such as project number, subproject number, system numbers, task number, etc.

9a. ORIGINATOR'S REPORT NUMBER(S): Enter the official report number by which the document will be identified and controlled by the originating activity. This number must be unique to this report.

9b. OTHER REPORT NUMBER(S): If the report has been assigned any other report numbers *(either by the originator or by the sponsor)*, also enter this number(s).

10. AVAILABILITY/LIMITATION NOTICES: Enter any limitations on further dissemination of the report, other than those imposed by security classification, using standard statements such as:

(1) "Qualified requesters may obtain copies of this report from DDC."

(2) "Foreign announcement and dissemination of this report by DDC is not authorized."

(3) "U. S. Government agencies may obtain copies of this report directly from DDC. Other qualified DDC users shall request through ______________________."

(4) "U. S. military agencies may obtain copies of this report directly from DDC. Other qualified users shall request through ______________________."

(5) "All distribution of this report is controlled. Qualified DDC users shall request through ______________________."

If the report has been furnished to the Office of Technical Services, Department of Commerce, for sale to the public, indicate this fact and enter the price, if known.

11. SUPPLEMENTARY NOTES: Use for additional explanatory notes.

12. SPONSORING MILITARY ACTIVITY: Enter the name of the departmental project office or laboratory sponsoring *(paying for)* the research and development. Include address.

13. ABSTRACT: Enter an abstract giving a brief and factual summary of the document indicative of the report, even though it may also appear elsewhere in the body of the technical report. If additional space is required, a continuation sheet shall be attached.

It is highly desirable that the abstract of classified reports be unclassified. Each paragraph of the abstract shall end with an indication of the military security classification of the information in the paragraph, represented as *(TS), (S), (C), or (U)*.

There is no limitation on the length of the abstract. However, the suggested length is from 150 to 225 words.

14. KEY WORDS: Key words are technically meaningful terms or short phrases that characterize a report and may be used as index entries for cataloging the report. Key words must be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context. The assignment of links, roles, and weights is optional.

13. Abstract (Concluded)

The most notable difference is the low wear rate which has been measured for pyrolytic samples in ultra-high vacuum. This absence of "dusting wear" accompanied by a moderate coefficient of friction for unannealed pyrolytic graphite is very interesting in regard to many possible applications. (U)

An optical microscope study of the surfaces resulting from bulk shear of graphite crystals is presented. The majority of new area produced apparently results from unresolved lamellar shear. However, some of the applied shear stress appears to be resolved in cleavage and also dissipated into deformation of the sample. Similar experiments with molybdenite yield a truer shearing action; comparisons are described. (U)